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Automated Nondestructive Evaluation Method for Characterizing Ceramic and Metallic Hot Gas Filters

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Introduction

Hot gas filtration is a technology to clean fuel gas from advanced coal combustion or gasification power conversion processes. One method to clean the fuel gas is to use rigid ceramic candle filters (1-3). For several years, monolithic oxide and non-oxide ceramic candle filters have shown the most promise because of their durability at aggressive high temperature operation environments (4,5). More recently, oxide/oxide composites as well as other material such as Fe-Al have also been developed. To date, almost all materials have demonstrated degradation in real and laboratory simulated operation environments (4). The degradation of the material manifests itself in a reduced strength and thus reliability of usage. Since as many as 3000 to 4000 of these filters are likely in a large plant, and since the costs for unscheduled down time in a plant can be very significant, it becomes imperative to develop nondestructive evaluation (NDE) methods to assess the condition of these filter elements.

Several non-destructive evaluation methods have been under development at Argonne National Laboratory for application to rigid ceramic hot gas filters. One that has shown the most promise is

the Acousto-Ultrasound (AU) method (6-10). Acousto-Ultrasound (AU) utilizes a combination of traditional ultrasonics and acoustic emission measurements. An ultrasonic pulse or elastic wave is introduced into the material by a transmitting transducer and a second transducer detects the transmitted elastic wave. Usually such a method is applied to plates and tubes. It is suggested that the wavelength of the transmitted pulse be carefully chosen to produce a wave such that the whole volume of material between the transducers is insonified. This is necessary because the wave propagation is affected by scattering from microstructural features like porosity, cracks, differences in elastic properties of phases present and mode conversion (9). Analysis of the detected signal allows determination of an AU parameter referred to as a Stress Wave Factor (SWF). Since many hot gas filter elements would need to be evaluated at any scheduled or unscheduled shut down, if the AU method could be shown to be an estimator of retained strength, then the AU method could be automated through use of special wheel transducers. In order to establish if a significant correlation could be established between the AU data and retained strength, a series of experiments were conducted on a wide range of materials including clay-bonded SiC, recrytallized SiC and oxide composites.

Objective

The objective of this work was to develop a nondestructive (NDE), cost-effective and reliable method to assess the condition of rigid ceramic hot gas filters. The work was intended to provide an end user, as well as filter producers, with a nondestructive method to assess the "quality" or status of the filters.

Approach

The approach used in this project was to utilize several filter material types with various real and simulated damage levels. The real damage levels were induced through plant service and the simulated damage levels were induced through various laboratory methods such as local thermal shock. While several NDE methods were evaluated on many filter material types, the first AU system developed used two 20 mm diameter 150kHz acoustic-emission transducers which were placed in direct contact with the hot gas filters. Load cells were used to allow establishment of the load being applied to each transducer so that crushing of the material locally was avoided. Figure 1 shows a schematic of the first set up. A computer was coupled to the pulse generator to control the pulse shape and strength. Previous work has shown that plate wave, as in this set up, will propagate as extensional or flexural waves as shown in Fig. 2.

Flexural waves travel at a slower speed than extensional waves. In all work, we use analysis of extensional waves. The stress wave factor from acousto-ultrasound data can be obtained in several different ways as noted in the ASTM guide (10). Two of these SWF methods are: (a) ringdown from amplitude-time acquisition, and (b) amplitude-frequency using Fourier transforms of the amplitude-time acquired data. Data using both methods have been studied. In the simple ringdown method a threshold must be set on the amplitude-time domain data and the number of peaks above that threshold established. Figure 3 shows examples of the detected pulses for both time-amplitude and Fourier transforms for alumina-mullite and clay-bonded SiC filters.

Project Description

This project was a cooperative project with Southern Research Institute (SoRI), Southern Company Services/Power Systems Development Facility and several industrial rigid ceramic hot gas filter

suppliers including Schumacher, Industrial Filter and Pump, Honeywell Composites (now GE Composites) and McDermott Inc. This project initially explored several NDE methods including infrared thermal imaging, high-speed x-ray tomography, air-coupled ultrasonics and acousto-ultrasonics. During the term of the work, cooperative efforts were established with the University of Tampere in Tampere, Finland. In this cooperative effort, staff of Tampare University supplied; a)- well-characterized hot gas filter test specimens that were exposed to a wide variety of laboratory environments, b)-complete microstructural analysis of these filter elements and c)- strength data for correlation to the NDE data. During the term of the project, the stress-wave factor data from the acousto-ultrasound method showed significant potential and thus received the most attention.

Results and Application

While tests were conducted on many different ceramic materials, for this paper, only tests conducted on two sets of monolithic hot gas filter samples and one set of oxide/oxide composite samples will be presented. In the first set of monolithic materials, both a clay-bonded SiC and an alumina-mullite were used. In the second set, only a clay-bonded SiC was used. The oxide/oxide ceramic samples were made using slurry coated Nextel N610 filaments wound using filament winding. Some of the oxide composite specimens were in the as-produced condition and some had seen service in a pilot plant. For the first sample set (4), the clay-bonded SiC samples were from a commercial grade material and were identified as S and the other was an alumina/mullite material and were identified as Q. For these tests, one sample of each type was exposed in air at 900°C for 2000 hours, later denoted as S1 and Q1. Two other samples, S2 and Q2 were exposed in air and water vapor at 900°C for 670 hours. Typically the samples used for exposure were 20-30 cm long segments cut from a filter with an outer diameter 60 mm and inner diameter 40 mm.

These samples were studied by momentary contact acousto-ultrasound using 150 kHz center frequency transducers. The two transducers were mounted on a "jig" so that they could be moved to different locations along the length of the 20-30 cm long sample. The transducers were spaced about 10 cm apart with the distance being set by the maximum detected amplitude. This spacing limited the number of SWF data points that could be obtained along the length of this sample set. The AU SWF data was different for each material and the data were compared only within single material type.

Stress wave factor data were acquired along the length of each sample at three angular locations and each time from several overlapping positions along the sample as noted in Fig. 4. Seven SWF pulses were averaged at each position. Later the samples were cut into rings each 20 mm in length for subsequent strength measurement using internal hydrostatic burst (11). Four strength specimens were associated with one SWF axial location using the average of three angular positions. The SWF used for analysis was the overall average of SWFs from all locations. Strength testing was done at room temperature and retained strength was calculated from the maximum pressure necessary to break the ring. The pressure was calculated from the maximum load as measured on the universal testing machine. Results were analyzed following the standard DIN 51 110 (11). The results of these experiments are shown in Table 1.

It is noted that the difference in SWFs and strength are very small and a strict reading of the data might suggest that the strength of samples S and Q of each filter are the same. Microstructrual analysis was carried out on these samples and one observation was that the porosity varied (5).

Open porosity and apparent density of selected specimens from several axes was studied by Archimedes' method with boiling water. Figure 5 shows that the porosity was far from uniform in these samples.

Analysis of the porosity variation porosity suggested that porosity variation is not significant for the SWF. A strong correlation between SWF and strength was established for filter S2. There was a significant variation in strength along the axis of filter S2 that corresponded with a large standard deviation in the overall SWF. The strong correlation between local SWF to local strength in S2 suggested that both SWF and strength are dominated by the same microstructural feature. Such a feature could be microcracking since there is not significant difference in phase content or composition of filters S1 and S2. For filter Q2 a large variation in porosity was found to exist along the filter. When the porosity data were averaged in the same manner as the SWF data, then again a correlation between porosity and SWF was found.

In the second set of monolithic samples, only clay-bonded SiC were studied. In addition, based on the evidence of a strong correlation between retained strength and SWF, the momentary contact transducer set up was replaced by an automated system utilizing wheel transducers. A schematic diagram is shown in Fig. 6 along with a photograph of the set up. In this set up, the wheel transducers were mounted in a fixture such that the hot gas filter, mounted in a "V"-shaped holder driven by computer-controlled stepper motors, moved under the wheels. A special software package was locally written, using LabView as the user interface, that constantly monitored and recorded the received wave forms with known location along the axis of the filter. The package not only controlled the speed at which the filter moved but also recorded the pressure on each wheel transducer through pressure transducers. This system provides a data acquisition that allows the filter to be moved with constant data acquisition at translational speeds of between 10 and 20 mm/ second. At 20 mm/second, a full length, 150mm long ceramic candle filter can be examined in less than 10 seconds. Assuming that 2 such devices would be at a plant site, and with wheel transducers located at two radial locations to allow for detection of radial variations, then 60-120 candle filters could be examined per hour. For a 3000 candle filter plant, with a 10-hour shift, the entire set of filters could be completely tested in 4-5 days.

For this second set of the experiments, using only clay-bonded SiC, three samples from each of two material sets, A and B, were examined. These were 50-cm long segments of hot gas filters. The intent of these six samples was to examine how well the AU method could track changes in retained strength. These six samples were first studied by the AU NDE method in the as- received condition. In any one three-sample set, one sample was kept as a reference, one was subjected to a treatment that consisted of a 500h soak at 850°C in a water vapor environment and the third sample was used for a 500 h hour soak at 850°C in a water vapor environment but with 8 heating/cooling cycles with the cooling cycle reaching below 100°C. The strength of the exposed samples was established after AU scans. Some problems occurred with the wheel transducers in the measurements of the samples used for the 500-hour soak at 850°C water vapor exposure. The strength data for the samples exposed to 500C with water vapor with heating/cooling exposure are not yet available.

Another aspect of this set of tests was to see if an energy parameter determined from the AU-data could provide a stronger correlation factor. The parameter chosen for study was a sum squared amplitude (SQA)(12) that can be described as

$$SQA = \sum_{j=0}^{N-1} \left| c_j \right|^2$$

where c_j is the amplitude of the signal from time domain data over a set axial length of the specimen. Figure 7 is one typical example of the constantly recorded SWF as well as the destructively measured hoop strength for one of the samples.

While the strength along the axis is uneventful, the SWF data also suggest that there is a rather uniform strength. By data from all the samples in this second set available at this time, that is the as- produced and water vapor exposed samples of both materials, the modulus of rupture strength and over all average of SWF data, a rather significant correlation is established as shown in Figure 8. It is noted that the materials A and B have different strength in as- received condition due to slight differences in the microstructure but the material type is essentially similar so the data can be used for single plot.

The last sample set studied was a set of oxide/oxide composites. The filters were again 60 mm outer diameter and 40 mm inner diameter. The producer had suggested that some of the powder used in processing had caused a lower-than-average strength in one of the samples. However, one filter of less than average desired strength in the as-produced condition was installed in the coal-fired test facility at the Power Systems Development Facility (PSDF) in Wilsonville, AL. This filter performed satisfactorily and was subsequently destructively analyzed for strength. The retained strength value when removed from service was higher than expected. The AU data obtained on this filter, without a prior knowledge of this strength, also showed a higher SWF value which, based on previous data, would suggest a higher strength. From the point of view of the AU methods, the wave form from the oxide materials is significantly different than the wave form from the monolithics. Figure 9 shows the detected wave form for this oxide composite material as well as a wave form. Using the ring-down count method for these composites is not as straight forward for such materials as there are clear harmonics. By setting the threshold high however, ring down can be used.

By using the AU data, correlations are obtained between SWF and hoop strength for the oxide/oxide materials were obtained as shown in Figure 10.

Summary and Future Work

An acousto-Ultrasound NDE method has been developed and studied as a method for estimating retained strength of various ceramic materials used as rigid ceramic candle filters. Both a momentary contact method and an automated method using wheel transducers have been developed. Data obtained from the Acousto-Ultrasound method, stress-wave factor, (SWF) were correlated with hoop strength for several candidate materials including: (a) alumina/mullite, (b) clay-bonded SiC and (c) oxide/oxide composites. In several experiments, including both laboratory controlled experiments and field tests, the correlation between the strength and the SWF holds. The SWF, a simple ring down count of amplitudes exceeding a preset threshold value of the detected ultrasound pulse, was determined for several locations along the samples. The variation in the local values of SWF was studied in terms of strength, porosity and microstructure of the samples. It was

found that the overall average of SWF correlates to the MOR of strength even when the change in MOR strength due to exposures was very small. The results suggest that local variations of porosity in alumina/mullite based filter could explain some of the variation of local SWF values along the filter samples but porosity does not determine the strength of the material. In clay bonded SiC based material exposed at high temperature air and water vapor the local SWF values followed the change in median strength of the sections.

The acousto-ultrasound method is capable to monitor the changes in strength of hot gas filters and some understanding of the microstructure-SWF-strength relation have been gained. Further work is needed to more firmly establish the factors that impact strength reduction of hot gas filter materials and how these features affect the SWF. Regardless of these other information, it seems that this method could be used for go/no-go type of decisions with the proviso that a baseline set of data are established for any particular filter material system. It is to be noted that the lamb wave transmitted through the material and the microstructure of the material are complicated. It is to be noted further that the exact failure mechanisms of the material are not known.

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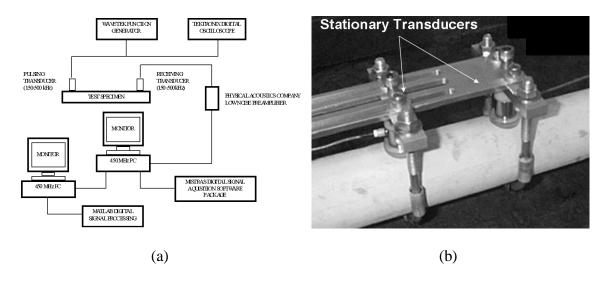


Fig. 1. AU System for NDE of Hot-Gas Filter: (a) Schematic Diagram, (b) Photo Showing Acousto-Ultrasound System in the Momentary Contact Configuration.

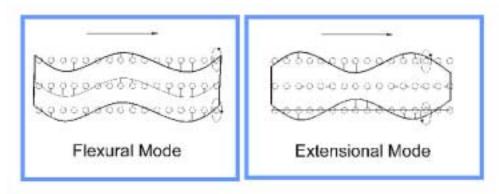


Fig. 2. Schematic Diagrams Showing Two Propagating Lamb Wave Modes.

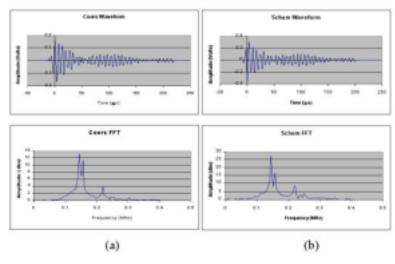


Fig. 3. Detected Wave Forms and their Fourier Transforms as a Function of Ceramic Material. (a) Alumina-Mullite, (b) Clay-Bonded SiC.

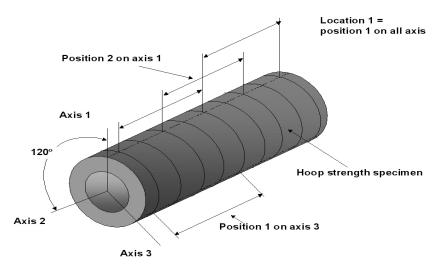


Fig. 4. Schematic Diagram Showing the Three Axial Positions Used for SWF Data Acquisition and Position of the Strength Specimens.

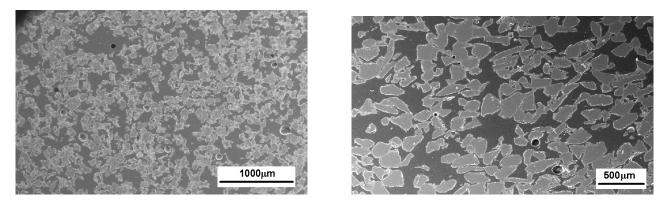


Fig. 5. Comparison of Open Porosity for the Two Materials, S on the Left and Q on the Right.

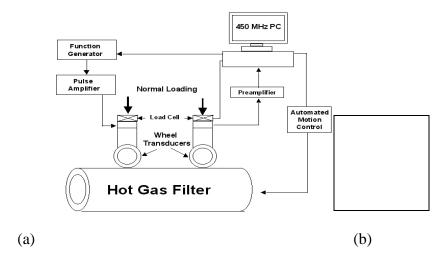


Figure 6. Automated Wheel Transducer AU Set Up for Fast Scanning. (a) Schematic Diagram, (b) Photo Showing Acousto-Ultrasound System in the Automated Configuration with the Wheel Transducers and Load Monitoring Load Cells.

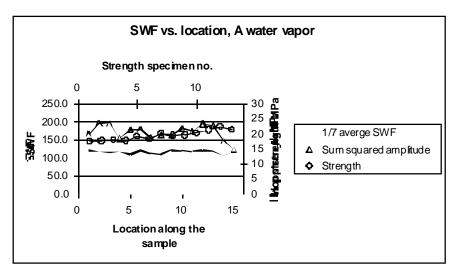


Fig. 7. Strength Profile, 1/7 of the Average of Original SWF and Average of Sum Squared Amplitude along the Filter Sample A after Exposure to High Temperature Water Vapor.

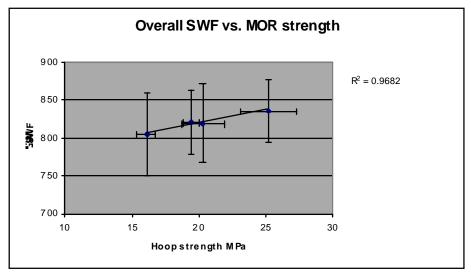


Fig. 8. Overall SWF vs. Modulus of Rupture Strength of the Samples.

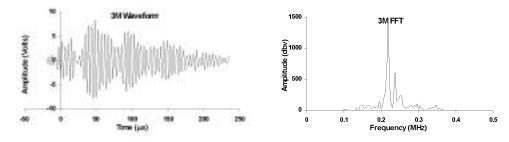


Fig. 9. Detected Wave Form and Corresponding Frequency Spectrum of Guided Plate Waves for Oxide/Oxide Ceramic Matrix Composite Systems.

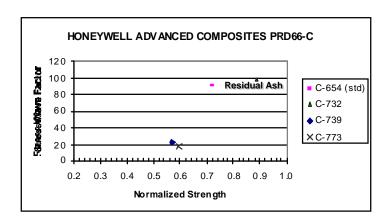


Fig. 10. Correlation of AU-Determined SWF Values Versus Hoop Strength for Filament Wound Oxide/Oxide Ceramic Filters

Table I. MOR Strength, its 90-% Confidence Range, SWF and its Standard Deviation Calculated from the Local SWF Values of the Studied Filter Samples.

Filter	MOR [MPa]	MOR (1-a=0.9)	SWF	SWF STDEV
Q1	18.5	17.7-19.4	263	5
Q2	18.1	17.6-18.6	257	17
S 1	17.8	16.9-18.1	231	13
S2	17.0	16.0-18.6	220	24